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EXTRACTION CONDUCTOMETRIC DETERMINATION OF SCANDIUM AND THORIUM IN KAOLINS

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Abstract

A method for extraction - conductometric determination of scandium and thorium with tributyl phosphate and trioctyl phosphine oxide in kaolin was developed. The equivalent electrical conductivity of the complex of scandium and thorium with 2,7-dinitrozo-1,8-dihydroxynaphthalene-3,6-disulfonic acid was found. A scheme for the separation and determination of kaolin content is proposed. The isotherms of scandium and thorium extraction are constructed. It was shown that at a concentration of nitric acid of 15.6 M they do not interfere with the determination of thorium and scandium.

Keywords: scandium, thorium, 2,7-dinitrozo-1,8-dihydroxynaphthalene-3,6-disulfonic acid, conductometric titration, extraction.

Introduction

Scandium and thorium are found together with iron and rare earth metals in various minerals, kaolins and waste from technological plants. According to its analytical properties, scandium and thorium adjoin the rare-earth elements very close to the earth; when a group of rare earths is distinguished, these two elements always turn out to be with them, this is due to their close similarity and complete isomorphism of all their salts [1].

The content of rare earth elements in apatites often reaches as much as percent; in very small quantities, rare earths are part of many rocks. The main source of rare earth mining is monocytes, its weathering product is monazite sand. Before the discovery of monazite, cerite was considered the main ore, then orthite. Rare earths themselves are of little use in industry (cerium is used together with thorium for gas-heating caps and is used for the manufacture of pyrophoric alloys). The value of rare earth ores is determined by the content of thorium in them. And also the waste from copper, lead, zinc metallurgical and kaolin plants contains from 160 to 250 g/t of scandium and thorium [2].

To extract scandium from rare-earth metals [3], two-phase aqueous systems of PEG-3000 (C_2H_7OH) – $NaNO_3(NaNO_3 + KSCN) - H_2O$ polyethylene glycol were proposed for the selective extraction of scandium nitrate and thiocyanate complexes, followed by its complexometric and spectrophotometric determination. When extracting scandium ions from rare-earth ($Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho,$

Er, Tm, Yb and *Lu*) metals [4], tributyl phosphate with molecular iodine and tributyl phosphate concentration were used 80-100%, the authors of [5] studied the extraction of scandium with diantipyrylmethane, diantipyrylbutane and diantipyrylheptane from naphthalene-2-sulfonic acid solutions in various types of systems, as well as without the use of an organic solvent.

In work [6] the interfacial distribution of micro-quantities of nitrates and perchlorates *Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb* and *Lu* by aqueous solutions of NH_4NO_3 or NH_4ClO_4 and solutions of phosphoryl substituted azapodand $[Ph_2P(O)CH_2CH_2OCH_2CH_2]_2NBu$ in dichloroethane was studied. Extraction methods of extraction and concentration of rare earth elements (REE) and scandium from solutions of mineral acids are proposed in the works [7], [8] used bi- and polydentate neutral organophosphate compounds - dioxides of methylendiphosphines [9], oxides (dialkylcarbamoylmethyl) of diarylphosphines (CMFO) [10], M-diphenylphosphoryl-N-alkyl urea [11], phosphorylated calixarenes [12]-[15], phosphoryl subshands [16], and tetraalkyl diglycol amides [17]-[19].

In recent years, contamination of the environment by radioactive elements is considered as one of the most important threats to human health, one of them is thorium [20], [21]. Thorium and its compounds are highly toxic, causing cancer [22] and kidney damage [23], [24]. Thorium migration is influenced by a number of factors - pH, colloid formation, etc. [25]. To reduce environmental pollution, on the one hand, and return thorium to the production cycle, it is possible to determine it using various adsorbents [26], [27]. A number of methods were used to remove thorium from radioactive wastewater: chemical deposition [28], membrane process [29], ion exchange [30], liquid extraction [31] and adsorption [32]. The choice of a method is determined by the initial content of thorium, as well as the efficiency of the process, time, ease of implementation and cost [33]. In [34], uncharged molecules - biosorbents-were used instead of sorbents. To improve the sorption properties of biosorbents, various polymers or their mixture with minerals were used [35]. In [36] the ability of phosphate, carboxyl, amide, amine and sulfohydryl groups to form complexes with metal ions is shown.

To solve the urgent problems raised, it is necessary to develop a new modern method for determining small and micro concentrations of scandium and thorium with high metrological characteristics and analytical parameters. The paper proposes an extraction-conductometric method of determination. Conductometric method of analysis in comparison with other methods chosen by us differ in some advantages. It allows to carry out determination not only in transparent, but also in the painted and turbid solutions, and also in the presence of oxidizers, reducing agents and complexing organic reagents without big expenses and efforts. Conductometric methods allow to conduct research not only in water, but also in non-aqueous, mixed and water-organic solutions, where it is relatively easy to automate titration processes; to avoid separation of impurities, which usually interfere with determination by other methods, to produce differentiated titration of electrolyte mixtures, which cannot be realized by other methods [37].

1 Experimental part

When performing the experiment, 0.1 M of solutions of scandium and thorium were used for the preparation, which took their nitrate salt, dissolved in water and brought the total volume to the mark with bidistillate in a volumetric flask (100 ml). The exact concentration of metals was determined by a known technique [38].

A 0.1% solution of 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid was used as a titrant.

All reagents used were Used equipment, metals, reagents and solutions. For the titration of scandium and thorium with a solution of 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acids in order to establish the optimal pH values of the medium, the universal EV - 74 ionometer and the pH meter pH/mV/temr Meter P25 EcoMet of Korean production were used. In the work were used conductometer "MetlerToledo" and microfurette first class accuracy.

Qualified "OS.C.", "CH. C." and "C. F. A.". Standard solutions of thorium and other metals used in the work with a concentration of 1.0 mg / dm³ or less were prepared by dissolving their nitric acid salts in bidistilled water. Working solutions of the used metals were obtained by successive dilution of their initial strong solutions before the experiment.

2 Results and discussion

As a rule, during conductometric titration there is a change in the total electrolyte concentration of the analyzed solution, respectively, and the electrical conductivity of the entire system. When preparing the analyzed solution, different in nature and concentrations of background electrolytes or buffer mixtures are introduced into it, causing high electrical conductivity of the system under study, intended for quenching migration and adsorption currents, better mass transfer (transport) and other factors affecting the course, results and other physico-chemical parameters of conductometric titration [39].

To determine the stability constant of scandium, yttrium, europium, praseodymium, cerium and thorium complexes with β -hydroxyethylmaleinimide, 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid, the effect of concentration on the equivalent electrical conductivity was studied by the conductometric method.

In conductometry, to determine the dissociation constant of a weak electrolyte, the " ϵ " of a number of solutions are measured, their equivalent electrical conductivity, $\lambda = \frac{\epsilon \cdot 10^{-3}}{C_H}$ the degree of dissociation $\alpha = \frac{\lambda}{\lambda_0}$ and $\check{E}_{diss} = \tilde{n}\alpha^2 1 - \alpha$. Or use the equation obtained $K_{diss} = \frac{\lambda^2 \tilde{n}}{\lambda_0(\lambda_0 - \lambda)}$ from the previous one. The equivalent conductivity of the solution is calculated from reference data $\lambda_0 = \lambda_{0+} + \lambda_{0-}[\dots]$.

The calculated practical C_{diss} depends on concentration. Concentration-independent is the thermodynamic dissociation constant expressed in terms of ion and molecule activity.

$$K_{diss(a)} = \frac{\tilde{n}\alpha^2 f_{\pm}^2}{1 - \alpha}; \quad f_{\pm} = \sqrt{f_+ \cdot f_-}$$

for monovalent electrolyte.

The dependence of the specific conductivity on the concentration of the complex gives the equivalent conductivity of the complex, this is a qualitative characteristic of the complex, from it you can find the thermodynamic stability constant of the complex, which are shown in Fig.1.

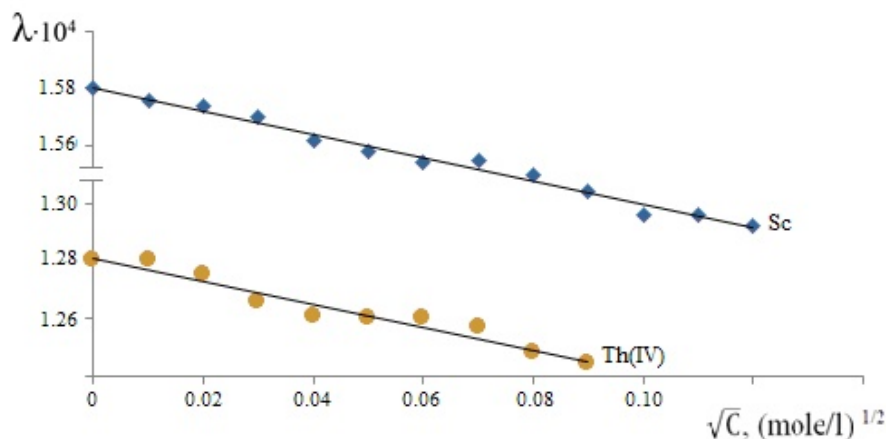


Fig. 1. The figure shows that the equivalent electrical conductivity of the complex is associated with the mobility of the complex in solution. The conductivity of the scandium complex is greater than that of thorium, which proves Goldschmidt's theory. According to this theory, the ionic radius of scandium is greater than that of thorium, so the stability constant is greater.

To establish the correctness and reproducibility of the developed conductometric method for determining scandium and thorium with a solution of 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid, it was necessary to titrate different concentrations of them with multiple parallel repetition. the data obtained are given in table 1.

From the data of table 1 it can be seen that the found concentrations of scandium and thorium are in strict accordance with the introduced quantities, which indicates the high correctness and reproducibility of the developed conductometric method for its determination with a relative standard deviation (Sr) not exceeding 0.261.

Using extraction, REE can be separated from each other, while the extractant must have a high viscosity, possibly higher flash point, low toxicity and low solubility, which contributes to the effective removal of the metal from the organic phase. In REM technology, various extraction systems are used and almost all major classes are used - neutral extractants, organic acids and organic bases [40].

Table 1

The results of conductometric titration of various amounts of scandium ions and the theory of solutions of 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid under optimized conditions ($P=0,95$, $n=5$)

Introduced Me, μg		Found, μg ($\bar{x} \pm \Delta X$)	S	Sr
Sc(III)				
1	0,45	0,46 \pm 0,13	0,12	0,261
2	0,90	0,97 \pm 0,18	0,16	0,165
3	1,80	1,88 \pm 0,28	0,25	0,133
4	4,50	4,54 \pm 0,32	0,28	0,061
5	9,00	9,15 \pm 0,26	0,23	0,025
Th(IV)				
1	1,16	1,25 \pm 0,17	0,15	0,120
2	11,60	11,67 \pm 0,2	0,18	0,015
3	23,20	23,27 \pm 0,3	0,26	0,011
4	46,40	46,33 \pm 0,36	0,32	0,007
5	92,80	92,68 \pm 0,48	0,42	0,004

Organophosphorus reagents are widely used as neutral extractants. Extraction of rare earth metals from neutral or slightly acidic nitrate solutions (tributyl phosphate (TBP), other neutral organophosphorus compounds) is carried out according to the mechanism of resolutely



For neutral and slightly acidic solutions (0.0001-0.05 mmol HNO_3) during TBF extraction, the concentration of metal nitrate in the organic phase increases with the growth of its aqueous phase and reaches 150-180 g/l [41] at a content of 300-400 g/l in the aqueous phase [42].

The method of extraction of scandium. Currently, the most widely used for the extraction of scandium are organophosphate derivatives of the class of phosphates $(RO)_3PO$, phosphanates $(RO)_2RPO$, Phosphinates $(RO)R_2PO$ and phosphinoxides R_3PO , which were used by us. as is known, in these compounds, the oxygen activity of the phosphoryl group increases in a number of: phosphate < phosphanate < phosphinate < phosphin oxide. In the same direction, we experimentally obtained results, where the bond strength of these compounds with scandium increases.

In order to increase the degree of scandium extractability and reduce the consumption of the ether used, it is first extracted from an acidic solution. As is known [43], in most cases scandium nitrate from acidic solutions (conc. HNO_3) is quite well extracted (95 – 100%) by TBP dissolved in chloroform. Scandium is extracted in

the form of compounds, solvated with three molecules of TBP. It is assumed that at HNO_3 concentrations in the aqueous phase (1 : 1), scandium extraction is carried out in the form of its salts with TBF, which subsequently pass into the organic phase. The distribution coefficient during extraction from 15.6 M HNO_3 is more than 1000, which allows quantitatively with a small error to separate scandium from lanthanides and other extraneous accompanying metals [44].

The main stage, during the analysis, is the destruction of the extracted scandium complex and excess extraction reagent sodium fluoride. Sodium fluoride quickly and completely destroys the extracted complex and itself does not participate in the reaction with 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid, titrated cation - scandium and does not participate in electrode processes [45].

To determine the degree of destruction of the extracted scandium complex and the excess of the extraction reagent, scandium was extracted with an extraction reagent - TBF dissolved in chloroform in the presence of different in nature and concentration of strong oxidants-potassium permanganate, hydrogen peroxide, chloric acid and chromium anhydride. The resulting mixture is heated to a boil and the complete destruction of the complex with a small excess of the extraction reagent. When the analyzed solution is cooled, a background electrolyte (universal b. b.) is introduced into it and titrate scandium with a solution of 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid. The results are shown in table. 2.

Table 2

**Results of extraction-conductometric determination 4.5 mg scandium
D=0.7 (P = 0.95; n=5)**

Extractant	Titant	Foreign cation; [x]	Introduced [x], μg	$\frac{[x]}{[Sc]}$	Found Sc(III), μg ; $(\bar{X} \pm \Delta X;)$	S	Sr
TBP (chlorophyll 1:1, 15,6 M HNO_3)	2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid	Tb(III)	4,9	1,08	4,52 \pm 0,22	0,19	0,042
		Gd(III)	12,0	2,66	4,61 \pm 0,39	0,34	0,073
		Pr(III)	13,5	3,00	4,55 \pm 0,35	0,31	0,068
		Ce(III)	14,4	3,20	4,58 \pm 0,12	0,11	0,024
		Er(III)	15,0	3,33	4,48 \pm 0,10	0,09	0,020

It is established that by means of hybrid extraction-conductometric titration it is possible to determine scandium with a solution of 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid in various by nature complex industrial materials and environmental objects.

The method of extraction of thorium. Deep purification of thorium compounds from silicon and other foreign interfering metals, complexing compounds of

anions forming stable complex compounds with thorium was carried out by decomposition of the analyzed sample with solutions of 1.0 M HNO_3 and 0.8 M $HClO_4$. After opening the sample, thorium was extracted with 0.1 M trioctyl phosphine oxide (TOFO) [46] prepared in toluene with a nitric acid and perchlorate mixture (1.0 M HNO_3 and 0.8 M $HClO_4$). Thorium extraction was carried out with a mixture containing aqueous and organic phases (VB :Vo) = (1: 10) for 30 minutes, repeated agitation of the analyzed sample in the separation funnel. After extraction, a (5.0 ml) organic phase is taken and 0.3 M H_2SO_4 is re-extracted for 10 min. the Experiments were carried out under normal conditions.

The selected nitrate-perchlorate thorium extraction with TOFO extraction reagent prepared in toluene allows to achieve 97-99% thorium extraction at 5-10 times its concentration [47].

The methodology of the analysis. The analyzed solution containing 0.01 M of thorium was stirred, shaken repeatedly and extracted with a dividing funnel 5.0 ml of 0.1 M TOFO solution in toluene for 25-30 minutes. after delamination, 5.0 ml of the organic phase was taken into a tube with a lapped cork (50.0 ml) and re-extracted 5.0-10.0 ml with 0.3 m H_2SO_4 Solution for 10 minutes. After such an operation, the organic phase is separated, and the re-extract is used to determine thorium. During re-extraction, which is especially necessary, a strict quantitative transition of thorium into the aqueous phase in one action should be ensured in order to reduce its losses to a minimum. To the aliquot part of the analyzed solution, a background electrolyte or a universal Britton-Robinson buffer is added and thorium is titrated with a solution of 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acid. The results of extraction-conductometric determination of thorium with a solution of 2,7-dinitroso-1,8-dihyde-roxynaphthalene-3,6-disulfonic acid are given in table. 3.

Table 3

**Results of extraction-conductometric determination 23.2 mg thorium
(D=0.78) (P = 0.95; n=5)**

Extractant	Titrat	Extrane ous cation; [x]	introdu ced[x], µg	$\frac{[x]}{[Th]}$	Foundh Th(VI), µg; ($\bar{X} \pm \Delta X$;))	S	Sr
TOFO in toluola 1:1 1,0 M HNO_3 and 0,8 M $HClO_4$	2,7-di- nitroso- 1,8- dihyde- rock- synaphthal ene-3,6- disul- phocic acid	Sc(III)	20,0	0,86	23,24±0,07	0,06	0,002
		Nd(III)	25,0	1,07	23,21±0,03	0,03	0,001
		Ho(III)	30,0	1,29	23,27±0,13	0,11	0,005
		Pr(III)	35,0	1,50	23,26±0,17	0,15	0,006
		Gd(III)	39,0	1,68	23,23±0,14	0,12	0,005
		Er(III)	46,0	1,98	23,25±0,16	0,14	0,006

It is established that with the help of the developed hybrid extraction-conductometric method it is possible to determine thorium with a solution of 2,7-dinitroso-1,8-dihydroxynaphthalene-3,6-disulfonic acids in various by nature complex industrial materials and environmental objects.

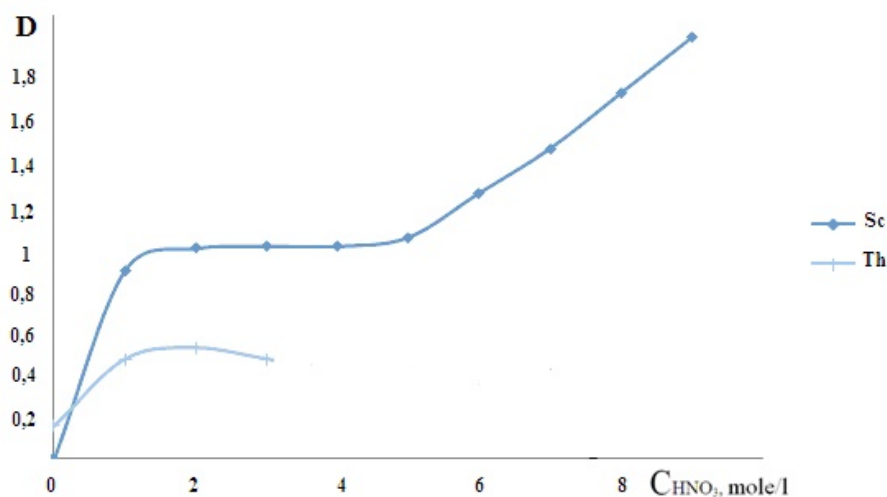
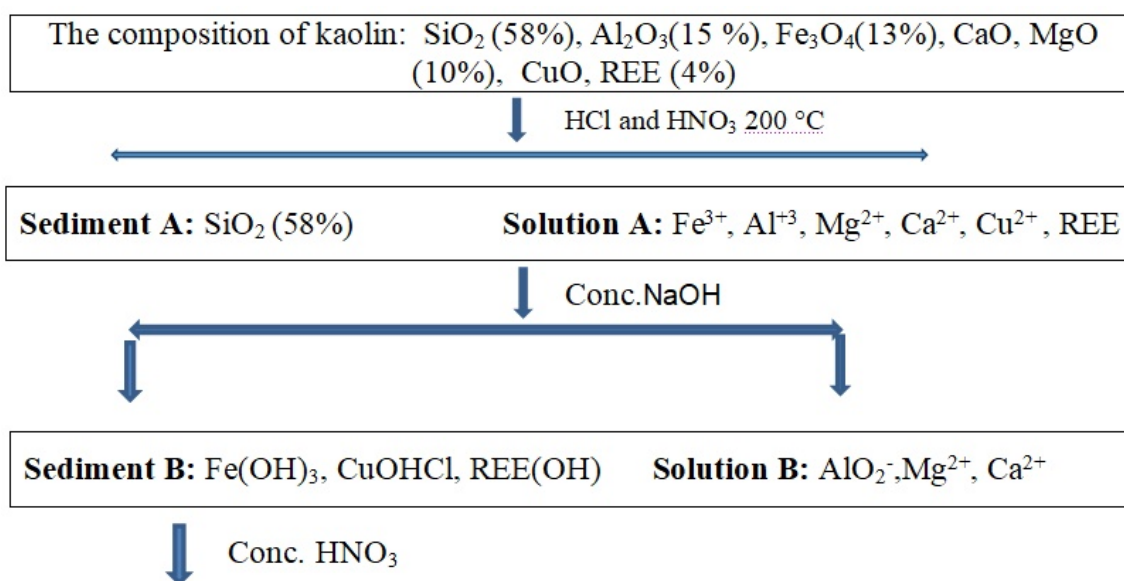
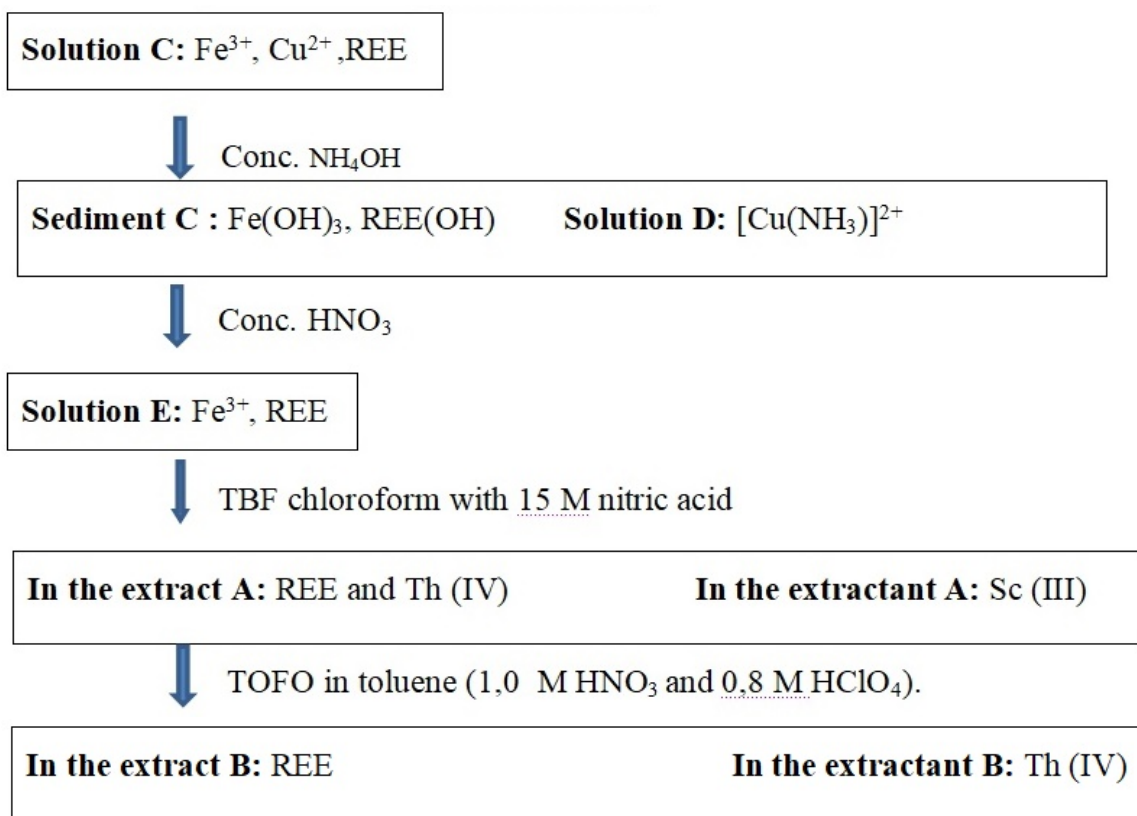


Fig.2. Isotherms of scandium and thorium extraction in nitric acid solution. From the figure it can be seen that when scandium and thorium are separated in a nitric acid solution, scandium is well separated from TAFF in an acidic medium.

Application of the developed method of determination of scandium and thorium in the composition of kaolin.

Separation of REE from kaolins





3 Conclusion

When measuring the concentration of scandium and thorium by conductometric titration, the yield of scandium and thorium was 98%, 0.12 mg/t of scandium and 0.34 mg/t of thorium.

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